Analysis of Sodium Hydrogen Carbonate

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Introduction:

Sodium hydrogen carbonate (NaHCO₃), also known as baking soda, is a chemical salt that can be used for both domestic and medicinal purposes. For example, when used in baking, it causes batter mixtures to rise. The sodium hydrogen carbonate reacts with H⁺ to form carbonic acid (H₂CO₃) which can readily bifurcate into water (H₂O) and carbon dioxide (CO₂).

$$NaHCO_3(aq) + H^+(aq) \rightarrow H_2CO_3(aq) + Na^+(aq)$$

 $H_2CO_3(aq) \rightarrow H_2O(l) + CO_2(g)$

The carbon dioxide produced from this reaction is trapped within the batter mixture and these trapped gases expand to allow the batter to rise (2).

Sodium hydrogen carbonate is also used in intravenous solution to treat metabolic acidosis (buildup of acid from metabolic processes), which if left untreated, induces shock and eventually death. Metabolic acidosis is caused by an imbalance of acids and bases within the body- the body either produces excess quantity of acids or the kidneys cannot neutralize enough acid which then begins to build up in the body (4). The pH in the bloodstream is regulated by a buffer of carbon dioxide, bicarbonate anion, and carbonic acid, that follows the equilibrium reaction:

$$CO_2(aq) + H_2O(l) \rightleftarrows H_2CO_3(aq) \rightleftarrows H^+(aq) + HCO_3^-(aq)$$
 (3)

When there is too much carbon dioxide in the bloodstream, the reaction leans to the right due to Le Chatelier's principle and the concentration of H⁺ ions increase, lowering the pH. If the

body does not produce enough bicarbonate anion, the reaction leans right and the concentration of H⁺ increases, lowering the pH. When intravenous sodium bicarbonate solution is injected into the bloodstream of a person diagnosed with acidosis, the increase in the amount of bicarbonate anion causes the reaction to lean to the right, decreasing the H⁺ concentration and bringing the pH back up to safe levels.

In both uses for sodium hydrogen carbonate, the purity of sample becomes a critical factor to consider. Too many impurities present in baking soda will decrease the amount of carbon dioxide produced. For an intravenous sodium hydrogen carbonate solution, the presence of sodium carbonate under a certain amount could positively impact the effectiveness of the solution in treating acidosis, dissociating into bicarbonate anion and sodium cation:

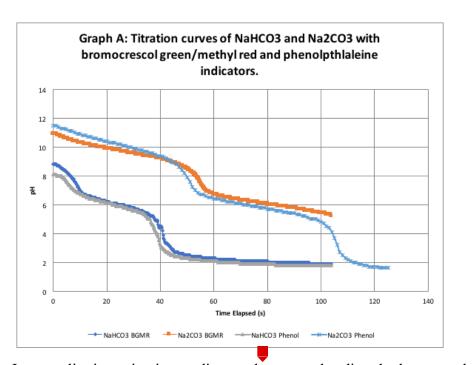
$$Na_2CO_3(aq) + 2 H^+(aq) \rightarrow 2 Na^+(aq) + HCO_3^-(aq) + H^+(aq) \rightarrow 2 Na^+(aq) + H_2CO_3(aq)$$

Each mole of sodium carbonate in the solution effectively acts as two moles of sodium hydrogen carbonate, so one must be very careful when sodium carbonate is present in significant amounts, as the dosage calculations must consider the sodium carbonate present in solution so as not to accidentally harm the patient via blood cell crenation nor alkalosis. It is not critical, having as pure a sample as possible is desirable since it allows for the simplest dosage calculations. With these uses in mind, the purity goal is no less than 90% sodium hydrogen carbonate by mass.

Experimental procedures

The sample of sodium hydrogen carbonate in this experiment was obtained from samples made earlier in the semester through the Solvay process involving solid carbon dioxide, saline

solution, and ammonia. Sodium hydrogen carbonate made in this way is likely to have contaminants from the products of intermediate reactions, which could consist of sodium chloride, ammonium chloride, water, and sodium carbonate. This determined the direction of our experimental procedure.



In an earlier investigation, sodium carbonate and sodium hydrogen carbonate were both titrated with hydrochloric acid using phenolphthalein and bromocresol green/methyl red indicators separately. The color of the indicator became clear/grey at the first equivalence point of both solutions (refer to Graph A), however the sodium carbonate solution had a higher initial pH than the sodium hydrogen carbonate solution, making the pink to grey transition more distinct. The phenolphthalein indicator was visible in both solutions before titration and remained visible through the first equivalence point, and the indicator turned to clear/grey at the second equivalence point. This meant that the amount of sodium carbonate present in the sample could be determined by titrating the sample in solution with hydrochloric acid using phenolphthalein indicator, and the amount of sodium hydrogen carbonate present in the sample

could be determined by subtracting the volume of titrant used at the first endpoint from the volume of titrant used at the second endpoint based on the titration equations equations:

$$Na_2CO_3(aq) + 2 HCl(aq) \rightarrow 2 Na^+(aq) + HCO_3^-(aq) + HCl(aq) \rightarrow 2 NaCl(aq) + 2 H_2CO_3(aq)$$

 $NaHCO_3(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2CO_3(aq)$

To analyze the composition of sodium hydrogen carbonate and sodium carbonate using titrations, 0.3 grams of sample were weighed and dissolved with DI water in an Erlenmeyer flask. Phenolphthalein indicator was added and the solution was titrated with 0.1000M HCl to the first equivalence point. Bromocresol green/methyl red indicator was added, and the solution was titrated to the second equivalence point. The solution was boiled to achieve a sharper endpoint, and then was carefully titrated to completion. This test was done thrice (see Table 1).

To determine the mass of chloride present in the sample, silver chloride precipitate was formed in a double displacement reaction and then weighed. 0.25g of sample were weighed to the nearest 0.0001g and added to a beaker. 50 mL of DI water were added to the beaker to dissolve the sample. Nitric acid was added to the beaker to protonate sodium carbonate and sodium hydrogen carbonate so that silver nitrate could not react with them. Acid was added until bubbles stopped forming. Since the amount of chloride ions present in the sample was undetermined, an excess volume of 0.1M silver nitrate of about 20 mL were added to the solution. The mixture was then boiled with Bunsen burner for roughly 2 minutes to coagulate the smaller silver chloride particles into filterable clumps. A dry filter crucible was weighed to the nearest 0.0001g and the mixture was filtered through this crucible under a vacuum (Lab manual, Section V, Appendix III). The filtered precipitate was dried in an oven at 40°C for 15 minutes. It

was then taken out, let cool, weighed, and put back in until the weight no longer decreased. The moles of silver chloride with the mass of the sample determined how many chloride ions were present in the sample. This test was done twice (see Table 2).

To verify if ammonium chloride is present in the sample, 0.50 grams of the sodium hydrogen carbonate sample was weighed out to the nearest 0.0001g and added to a beaker. It was then dissolved using roughly 20 mL of deionized water. 10 mL of 0.1M NaOH was added to the solution so that it could convert any of ammonium cation into ammonia. The beaker was wafted to smell for any signs of ammonia. This test was done once. The ammonia smell would indicate presence of ammonium ion in the sample. If ammonia smell is present, this means that there is some ammonium cation in our sample, meaning that the percent composition of ammonium chloride would have to be considered in the total composition of the sample.

The water test analyzed if the mass of the sodium hydrogen carbonate sample was affected by presence of water molecules. 0.25g of the sodium hydrogen carbonate were weighed and added to the beaker. The beaker containing the sample was then weighed to the nearest 0.0001g and then placed into an oven of 40°C. Between 5 minute intervals the beaker was taken out, cooled for 30 seconds, and then measured to detect whether the mass decreased or remains the same. This process was continued until the mass of the beaker containing the sodium hydrogen carbonate sample does not change. Three water tests were taken of the sample. (see Table 3)

Tabulated Results

Table 1: Percent of sodium carbonate and of sodium hydrogen carbonate in sample						
Mass of Sample (g)	V _i (initial volume of HCl in titration stand) (mL)	V ₁ (volume of HCl in titration stand at Na ₂ CO ₃ endpoint) (mL)	V ₂ (volume of HCl in titration stand at NaHCO ₃ endpoint) (mL)	%Na ₂ CO ₃	%NaHCO ₃	
0.3005	1.50	3.80	37.60	8.11	88.00	
0.3001	2.39	4.45	38.60	7.28	89.83	
0.3008	4.51	7.68	40.60	11.17	83.08	
0.3035	4.75	8.00	41.24	11.35	83.01	
% Mean:			9.48	85.98		
% Relative Average Deviation:			18.81	3.41		

Table 2: Percent Chloride in sample					
Mass of Sample (g) ±0.0001g	Mass of AgCl precipitate (g)	% Cl ⁻			
0.2511	0.0241	2.375			
0.2516	0.0195	1.919			
% Mean:	2.148				
% Relative Average Deviation:	10.649				

Table 3: Percent water in sample				
Weight of sample before	Weight of sample after	% H ₂ O		

31.2189	31.1351	0.2684
65.2208	65.1350	0.1316
81.1072	81.0085	0.1217
% Mean:	0.1739	
% Relative Average Deviation	36.2438	

Discussion

The percent composition sodium hydrogen carbonate in the sample was determined to be 85.98% with a relative average deviation of 3.413%. The percent composition of chloride in the sample was determined to be 2.148% with a relative average deviation of 10.65%. The percent composition of water in the sample was determined to be 0.1739% with a relative average deviation of 36.24%. The percent composition of sodium carbonate in the sample was determined to be 9.477% with a relative average deviation of 18.81%. These values did not meet the initial purity goal of >90% and with all of the relative average deviations having values >0.3%, the percent compositions are not precise enough to assess the true composition accurately.

Even though the sample was ground with a mortar and pestle, it is possible that the distribution of molecules in the sample was not homogenous. It is also notable that the first two titrations were done on the first day, and the last two were done on the second day. These first two titrations gave very different values for the percent composition of sodium hydrogen carbonate and sodium carbonate, however the second two titrations had percent compositions that had a relative average deviation of >0.1% relative to each other, and were much less than the

percent compositions from the titrations of the first day. This is most likely to have been caused by the concentration of the hydrochloric acid being unequally distributed in solution during the first set of titrations, and being much more equally distributed in solution during the second set of titrations. Another possibility, is that the distribution of possible contaminants within the sample was unequal within the sample, allowing for different "chunks" of sample with different percent compositions to be present in the total sample, and if one of these chunks were used in the titrations their distinct percent compositions would be reflected in the results, however this is much less likely due to the sample being ground prior to the use of any analytical methods and due to the consistency seen in the second set of titrations.

Within the chloride analysis, the reaction between the sample in aqueous solution and nitric acid to produce carbon dioxide and protonate all the carbonate and bicarbonate in the solution might not have been complete. It should be noted that that silver (I) carbonate is also insoluble, so carbonate or bicarbonate of the sample must be protonated so that it doesn't form Ag₂CO₃. Not adding enough nitric acid so that the carbonate or bicarbonate was all protonated, could have yielded above average mass of the precipitate and lowered the purity of the sample.

Measurements of the water test varied greatly by >36% deviation. This is primarily due to the change in mass between immediate extraction from the oven to around 1 minute after. Before it was understood that such difference existed, the execution of the first water test took roughly 45 minutes whereas the other two took roughly 20 minutes. Measurement of the sample promptly after it had been in the oven for an interval of time displayed lower mass, but the measurement should have been taken after a short period of cooling down. These variances might have impacted the % water composition and contributed to the wide deviations.

The test for ammonium in the sample was negative, so all the chloride detected from the chloride test was assumed to be from sodium chloride present in the sample.

Conclusion

The results of this experiment reveal the shortfall of laboratory synthesized sodium hydrogen carbonate for applications in domestic settings like baking and for remedial purposes like intravenous sodium hydrogen carbonate. These four analysis tests demonstrated: a) percent composition of sodium carbonate and sodium hydrogen carbonate present within the sample via volumetric analysis or titration with hydrochloric acid, b) percent composition of chloride ions within the sample, c) if ammonia ion was present in the sample, and d) a rough estimate of the mass of water that contributed to the overall mass of the sodium hydrogen carbonate sample. Although the % NaHCO₃ failed to reach the 90% critical value, the 85.98% composition still relatively close to 90%.

To use the sample in either baking or as an intravenous solution, the purity of sample would have to be >90%, so our sample cannot be considered for these uses. Typical baking soda has a purity of ~99% (1), so the sample would not perform well if used in baking. However, were the entire sample heated so that all sodium hydrogen carbonate decomposed into sodium carbonate, a relatively pure sample of sodium carbonate could be formed. Sodium carbonate has been used intravenously to buffer changes to blood pH caused by carbon dioxide (6). Assuming the sample of sodium carbonate was of good quality after heating, it could be used as an alternative for intravenous sodium hydrogen carbonate in an emergency to treat acidosis. This investigation introduced the process of analysis on the sodium hydrogen carbonate prepared via the Solvay process. The analysis tests indicate presence of above average percent impurities

within the laboratory synthesized sample. As such, it would require more quality lab control or an alternative mode (i.e. the mining of the trona ore) to generate a purer sample of sodium hydrogen carbonate.

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Appendix

$$\%NaHCO_{3} = \frac{\left((V_{2} - V_{1}) - (V_{1} - V_{0})\right)L * 0.1000M \; HCl * \; 84.006 \frac{grams \; NaHCO_{3}}{moles \; NaHCO_{3}}}{sample \; mass(g)} * 100\%$$

$$\%Na_{2}CO_{3} = \frac{(V_{1} - V_{0})(L) * 0.1000M \; HCl * \; 105.988 \frac{grams \; Na_{2}CO_{3}}{moles \; Na_{2}CO_{3}}}{sample \; mass(g)} * 100\%$$

$$\%H_2O = \frac{sample\ mass_{final} - sample\ mass_{inital}}{sample\ mass_{inital}} * 100\%$$

$$\%NaCl = \frac{\left(\frac{filter\; mass_{final} - filter\; mass_{initial}}{143.321\frac{grams\; AgCl}{mol \sqcup s\; AgCl}}\right)mol*\; 35.488\frac{grams\; NaCl}{moles\; NaCl}}{sample\; mass(g)} * 100\%$$

$$mean \ \% composition \ (\bar{x}) = \frac{\sum_{i}^{n} \% composition_{i}}{n}$$

$$relative \ average \ deviation = \frac{\left(\frac{\sum_{i}^{n}|\%composition_{i} - \bar{x}|}{n}\right)}{\bar{x}} * 100\%$$